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(54) Detergent tablet having a coating comprising carbamide

(57) The present invention provides detergent tablets which are at least partially coated with substances that contain carbamide. In a second aspect, the present invention provides a provides a process for making coated detergent tablets wherein said coating comprises carbamide and is applied in its liquid phase.

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Description

Field of the invention

[0001] The invention relates to solid block detergents made of compacted detergent powder, so-called detergent tablets, and especially relates to those tablets used in washing machines or dish washing machines which are at least partially coated by other agents as the ones used as detergents.

Background and Prior Art

[0002] Detergent compositions in solid block forms are well known in the art and described for instance in GB-A-0989683 and in GB-A-0911204.

[0003] Detergent tablets have several advantages over liquid, powdered or granulated products: They are easier to handle and to dispense into the washload or to insert into a water spray detergent dispenser. They use up less storage room because they are compressed. Further, separation of the various components of the detergent for example during the transport is avoided.

[0004] Nevertheless, some difficulties have been encountered with solely compressed compositions: Abrasion or even breakage of the tablet can occur during shipping and handling and thus loss of detergent material. To make the surface of the tablet hard enough the appliance of various coatings has been proposed. GB-A-0 989 683, published on 22nd April 1965, discloses a water-soluble organic film-forming polymer (for example, polyvinyl alcohol) as a coating. EP-A-0 716 144, published on 12th June 1996, also discloses laundry detergent tablets with water-soluble coatings which may be organic polymers including acrylic/maleic co-polymer, polyethylene glycol, PVPVA, and sugar.

[0005] Further coating materials have been proposed. EP-A-0 002 293, published on 13th June 1979, discloses a tablet coating comprising hydrated salt such as acetate, metaborate, orthophosphate, tartrate, and sulphate. EP-A-0 846 754, published on the 10th of June 1998, provides a tablet having a coating comprising a dicarboxylic acid. EP-A-0 846 755, published on the 10th of June 1998, provides a tablet having a coating comprising a material insoluble in water at 25°C, such as C12-C22 fatty acids, adipic acid or C8-C13 dicarboxylic acids.

[0006] Coatings have also been found to protect alkaline detergent compositions which are hygroscopic from absorbing substantial quantities of water from the atmospheric humidity during storage. EP-A-0 737 245, published on 6th July 1995, discloses a hydrophobic coating, including wax, fatty acid, fatty acid amides, and polyethylene glycol.

[0007] Additional ingredients for the coating have further been suggested. EP-A-0 846 756, published on the 10th of June 1998, provides a tablet having a coating comprising a disintegrant material and preferably an effervescent material to increase the dissolution of the coating upon contact with water.

[0008] With the different coatings suggested in the art, various problems arise as will be explained below.

[0009] Some coating material show a low solubility during the washing cycle and thus possible undissolved resins can occur. They can have a deleterious effect on the disintegration of the tablet.

[0010] The appliance of the coating materials causes additional complexity during manufacture. Sometimes it is necessary to use solvents which are difficult to remove. If the coating is applied from aqueous solution, the excess water has to be removed which requires a long heating time. Another disadvantage is that excess water can migrate into the tablet and affect the properties, especially solubility and storage stability. If the coating material is applied in the liquid phase, the coating material must be able to melt with substantial degradation.

[0011] Some coating materials such as the acidic components above can even negatively impact the detergency performance such as by lowering the pH.

[0012] In the field of washing detergents foaming can be a problem. Some of the above named coatings contain agents such as polyethylene glycol that have sufficient surface activity to produce excess foaming.

[0013] Another disadvantage of some coating materials is their price and availability.

[0014] It is an object of the present invention to provide a detergent tablet with a coating which does not suffer from the problems discussed above.

[0015] It is a further object of the invention to provide a coating material that can be applied in its liquid state.

[0016] It is a further object of the present invention to provide a coating material that contributes to the performance of the coated detergent composition.

[0017] It is a further object of the present invention to provide a coating material that readily dissolves in water.

[0018] It is a further object of the present invention to provide a coating material that is compatible with the environment including the respective legal requirements.

[0019] It is a further object of the present invention to provide a coating material that makes the detergent tablet hard enough to withstand abrasion during transport and usage.

Summary of the invention

[0020] The present invention provides a detergent tablet comprising a core and a coating, said coating covering at least a portion of the exterior surface of said core characterized in that the coating comprises carbamide.

[0021] The present invention further provides a detergent tablet wherein the coating contains carbamide and an agent that reduces the melting point of carbamide.

[0022] The present invention further provides a deter-

gent tablet wherein the coating comprises carbamide and a tenside.

[0023] The present invention further provides a detergent tablet wherein said agent that reduces the melting point of carbamide co-crystallises with said carbamide.

[0024] The present invention further provides a detergent tablet wherein the agent that reduces the melting point of carbamide is a salt.

[0025] The present invention further provides a process for making a coated solid detergent tablet comprising the steps of

- providing a particulate detergent composition
- compacting said detergent composition into tablets
- providing a coating material in its liquid phase, said coating material comprising carbamide
- applying said coating material to at least a portion of the exterior surface of said detergent tablet
- solidifying said coating material on said exterior surface of said tablet

[0026] The present invention further provides a process wherein said coating material comprises at least 50% carbamide.

[0027] The present invention further provides a process wherein coating material comprises carbamide and an agent that reduces the melting point of carbamide.

[0028] The present invention further provides a process wherein said agent that reduces the melting point of carbamide co-crystallises with said carbamide.

[0029] The present invention further provides a process wherein said agent that reduces the melting point of Carbamide is a salt.

Detailed description of the invention

[0030] The present invention provides solid detergents which can generally take the form of a coated tablet.

Detergent composition

[0031] Detergent compositions and in particular solid detergent compositions suitable for being coated with the coating material of the present invention are well known in the art. Such compositions may generally include without being limited to the following ingredients: surfactants, builders, bleaches, enzymes, chelating agents, soil release agents, soil anti-redeposition agents, dispersing agents, brighteners, suds suppressors, fabric softeners, dye transfer inhibition agents, perfumes, and the like.

[0032] It is to be noted that the presence of a coating in the tablet of the present invention substantially reduces the mechanical requirements for the solid detergent compositions comprised in the core.

Detergent tablet

[0033] Tablets of solid detergent compositions are well known in the art. There exist a wide variety of processes which are suitable for first manufacturing the solid detergent composition and subsequently for producing tablets from the solid detergent.

[0034] Solid detergent tablets can generally be made by mixing the ingredients including for example builder and surfactant. A exemplary and well known process for making particulate materials is spray drying leading to particulate materials of low bulk densities. Other suitable processes include granulation, fluid bed processes, compaction processes, extrusion, chemical processes (flocculation, crystallization, sintering, and the like), and the like. Subsequently, the particulate materials may be mixed with each other by a conventional mixing process well known in the art. Mixing can be carried out in a continuous process or in a batch process and can be carried out in a single process or in a plurality of sequential or parallel processes. Finally, detergent tablets can be made by any conventional compacting process such as tabletting, briquetting, extrusion, and the like.

[0035] Tablets suitable for being coated with the coating material of the present invention preferably have a diameter of at least 10, more preferably 20mm, yet more preferably 40mm. The diameter of the tablet of course depends on other factors such as the amount of solid detergent needed. Preferably, the amount of detergent in the tablet is such that one or few tablets provide the amount of detergent needed for the desired use. Preferably, the tablet has a weight of at least 10g, more preferably 20g, yet more preferably 30g.

Coating material

[0036] In general, coating a detergent tablet leaves many possibilities for formulating the detergent, many of these possibilities not being available without a coating. In particular the requirements for the mechanical properties such as the solidity and proneness to mechanical abrasion are significantly reduced. In addition, the interaction of the detergent with the environment such as the absorption of water from the air under normal storage are avoided at least to a large extent. A coating may for example can render a separate individual packaging of a detergent tablet superfluous.

[0037] Coating materials are particularly advantageous for multi-layer tablet cores, whereby the mechanical characteristics of a more elastic layer can be transmitted via the coating to the rest of the tablet, thus combining the advantage of the coating with the advantage of the more elastic layer. Indeed, mechanical constraints will be transmitted through the coating, thus improving mechanical integrity of the tablet.

[0038] The coating of the present invention is preferably made strong enough so that moderate mechanical shocks to which the tablets are subjected during han-

dling, packing and shipping result in no more than very low levels of breakage or attrition. Finally the coating is preferably made sufficiently brittle so that the tablet breaks up quickly when subjected to stronger mechanical shock. The mechanical properties of the coating material of the present invention can be adjusted by changing the thickness of the coating material and by including additional ingredients which impact the structure of the coating material as describe below.

[0039] In general, the coating material of the present invention can be present in any desired thickness and weight proportion compared to tablet core. Preferably, the coating forms from 1% to 10%, preferably from 1.5% to 5%, of the tablet weight.

[0040] Where the coating material of the present invention is intended to suppress interaction of the detergent with the environment or for other reasons, the coating material may be applied preferably to the entire exterior surface of the detergent tablet. Alternatively, where the coating is only intended to improve mechanical stability to the tablet, it may be sufficient to only coat a portion of the exterior surface of the tablet. The regions which are particularly prone to become affected by defects caused by external forces are the edges and the most importantly the corners of the tablet. Hence, in some embodiments of the detergent tablet of the present invention it is sufficient to only coat the corners and/or edges of the tablet with the coating material of the present invention.

[0041] The coating material of the present invention comprises at least 50% by weight, more preferably 60% by weight, yet more preferably 70% by weight, most preferably 80% by weight of carbamide. The mechanical properties of the crystallised carbamide render it suitable for being used as a coating material for detergent tablets. Carbamide readily dissolves in water and no environmental issues are associated with it.

[0042] In order to not interfere with the performance of the coated detergent composition, it is preferred that the aqueous solution of a the coating material of the present invention has a pH of at least 7. It is therefore preferred that the coating composition of the present invention comprises less than 10%, more preferably less than 5%, more preferably less than 1%, most preferably 0% by weight of an acidic component, the pH of a aqueous solution of such component being less than 7. In general, if additional ingredients for the coating material are chosen, it is preferred that substances are chosen which exhibit a pH of at least 7 in aqueous solution.

[0043] The coating material of the present invention generally can be applied in a number of ways. Two particularly suitable coating methods are a) coating with a molten material and b) coating with a solution of the coating material. For the coating material of the present invention, process a) is preferred for cost, energy consumption, and complexity reasons.

[0044] In a), the coating material is applied at a temperature above its melting point, and solidifies on the tablet. In b), the coating is applied as a solution, the solvent being evaporated to leave a coherent coating. The coating material can be applied to the tablet by, for example, spraying or dipping. Normally when the molten material is sprayed on to the tablet, it will rapidly solidify to form a coherent coating. When tablets are dipped into the molten material and then removed, the rapid cooling again causes rapid solidification of the coating material.

[0045] In order for the coating of the present invention to be applied to the tablet according to process a), the material must be heated above its melting temperature. By "melting temperature" is meant the temperature at which the material when heated slowly in, for example, a capillary tube becomes a clear liquid. At the same time, the coating material must not be heated to a temperature at which any constituent of the composition undergoes substantial degradation. In the case of carbamide, the degradation temperature is at about 120 °C and the melting temperature is about 132 °C.

[0046] The coating of the present invention preferably comprises an agent for reducing the melting temperature of carbamide. It is believed that such agents are effective by introducing a certain degree of disorder into the solid carbamide. Suitable melting point reduction agents include tensides such as those suitable for the detergent composition itself. An exemplary tenside is commercially available from FLUKA under the designation Span 20. Tensides incorporated into the coating material of course continue to contribute to the functionality of the detergent composition.

[0047] In order to not interfere with the mechanical properties of the coating to an undesirable degree, it is preferred that the melting point reduction agent forms a clathrate with the carbamide, in other words that the melting point reduction agent co-crystallises with the carbamide. Suitable melting point reduction agents which form a clathrate with carbamide include for example salts, preferably salts of metals of group 1 - 13 of the periodic table, preferably salts of group 1 and 2, even more preferably magnesium salts and most preferably magnesium acetate.

[0048] Surprisingly, it has been found that the salts used as meting point reduction agents further reduce the tendency of the carbamide to lead to ammonia to be released into the air upon dissolving. This tendency has prevented carbamide to be used widely as an ingredient for solid detergents and in particular in higher concentrations.

[0049] Upon solidification on the exterior surface of the tablet, the coating material may be exposed to mechanical stressed caused by the solidification of the coating material itself (e.g. shrinkage upon cooling) and caused by the detergent core (e.g. tablet relaxation). In addition, additional mechanical stresses may be exerted onto the coated tablet during further processing and during storage and transport. A possible results of these mechanical stresses are imperfections in the structure of the coating material such as cracks, fractures, splitted

edges, abrasion, and the like. Optionally, the coating material of the present invention comprises a component which is liquid at 25°C. This component is expected to improve the mechanical properties of the coating material of the present invention such that it is better able to withstand the above mechanical stresses. The component which is liquid at 25°C is preferably added to the coating materials of the present invention in proportions of less than 10% by weight of the coating, more preferably less than 5% by weight, and most preferably of less than 3% by weight. The component which is liquid at 25°C is preferably added to the coating materials of the present invention in proportions of more than 0.1% by weight of the coating, more preferably more than 0.3% by weight, and most preferably of more than 0.5% by weight.

[0050] Nonetheless, it is preferred that the coating material generally exhibits a crystallised structure. In other words, the coating material of the present invention as a whole should be generally solid at ambient temperature and should exhibit a certain degree of order at least in some portions. Generally, ordered portions of the coating material can be detected by means of conventional crystallography techniques such as X-ray analysis. The areas exhibiting a certain degree of order are believed to add mechanical strength to the coating material. For the liquid component mentioned above, it is therefore preferred to not or only partially co-crystallise with carbamide and thus remains liquid at least to certain extent. Such a liquid will the contribute some flexibility to a otherwise crystalline coating material in order to withstand mechanical stresses. Various suitable substances which are liquid at 25°C are well known in the art. Particularly preferred are those substances which further contribute to the overall performance of the detergent composition such as silicone oil. Other suitable liquid substances include but are not limited to polyethylene glycols, thermal oil, silicone oil, paraffin, triacetin, perfumes or alkaline solutions.

[0051] Mechanical dismantling of the coating material of the present invention can be supported by including disintegrant agents into the coating material. Generally, disintegrant agents work by exhibiting a substantial volume increase upon contact with water, the volume increase leading to mechanical stress in the coating and finally to its fracture.. The disintegrant agent is preferably included in the coating material at a level of up to 30%, preferably between 5% and 20%, most preferably between 5 and 10% by weight. Suitable disintegrants are described in Handbook of Pharmaceutical Excipients (1986). Examples of suitable disintegrants include starch: natural, modified or pregelatinized starch, sodium starch gluconate; gum: agar gum, guar gum, locust bean gum, karaya gum, pectin gum, tragacanth gum; croscarmylose sodium, crospovidone, cellulose, carboxymethyl cellulose, algenic acid and its salts including sodium alginate, silicone dioxide, clay, polyvinylpyrrolidone, soy polysacharides, ion exchange resins, polymers containing cationic (e.g. quaternary ammonium) groups, amine-substituted polyacrylates, polymerised cationic amino acids such as poly-L-lysine, polyallylamine hydrochloride) and mixtures thereof.

[0052] It may further be advantageous to incorporate fibres into the coating material of the present invention. Such fibres reduce the brittleness of the coating to such a degree that the coating becomes more resistant to externally applies mechanical stresses. At the same time, the functionality of the coating material is not negatively affected. Preferably, the fibres included in the coating material of the present invention have a length of at least 100 μm, more preferably of at least 200 μm and most preferably of at least 250 μm. Shorter fibres are less efficient in achieving the desired effects. Preferably, the fibres included in the coating material of the present invention have a length of less than 500 μm, more preferably of less than 400 μm and most preferably of less than 350 μm. Longer fibres may interfere with the dispersion of the coating material in water. Suitable fibre materials include but are not limited to viscose rayon, natural nylon, synthetic nylon (polyamides types 6 and 6,6), acrylic, polyester, cotton and derivatives of cellulose such as CMCs. Most preferred is a cellulosic material available under the trade mark Solka-Floc™ from Fibers Sales & Development. The fibres are preferably added at a level of less than 5% by weight of the coating, more preferably less than 3% by weight. The fibres are preferably added at a level of more than 0.5% by weight of the coating, more preferably more than 1% by weight.

Coating process

[0053] It is another aspect of the present invention to provide a process for coating solid detergent tablets.

[0054] Generally, coating of cores of solid detergent is a technique well known in the art for improving the mechanical stability of a tablet. In general, coating a detergent tablet leaves many possibilities for formulating the detergent, many of these possibilities not being available without a coating.

[0055] For applying the coating material of the present invention, a coating process including a step of applying the coating material in its liquid phase is preferred. Processes in which the coating material is applied as a solution require the additional step of removing the solvent by evaporation leading to more complex as well as cost and energy intensive processes.

[0056] The substantially insoluble material can be applied to the tablet by, for example, spraying or dipping. A large variety of suitable equipment for melting the coating material and for applying the molten coating onto the exterior surface are well known in the art.

[0057] Normally when the molten material is sprayed on to the tablet, it will rapidly solidify to form a coherent coating. When tablets are dipped into the molten material and then removed, the rapid cooling again causes rapid solidification of the coating material.

[0058] Where the coating material is intended to suppress interaction of the detergent with the environment, the coating material is applied preferably to the entire exterior surface of the detergent tablet.

[0059] Alternatively, where the coating is only intended to improve mechanical stability to the tablet, it may be sufficient to only coat a portion of the exterior surface of the tablet. The regions which are particularly prone to become affected by defects caused by external forces are the edges and the most importantly the corners of the tablet. Hence, in some embodiments of the process of the present invention it is sufficient to only coat the corners and/or edges of the tablet with the coating material of the present invention. Suitable processes and suitable equipment for this purpose are also well known in the art.

Claims

1. A detergent tablet comprising a core and a coating, said coating covering at least a portion of the exterior surface of said core **characterized in that** the coating comprises carbamide.

2. A detergent tablet according to claim 1, wherein the coating contains carbamide and an agent that reduces the melting point of carbamide.

3. A detergent tablet according to claim 1, wherein the agent that reduces the melting point of carbamide is a tenside.

4. A detergent tablet according to claim 3, wherein said agent that reduces the melting point of carbamide co-crystallises with said carbamide.

5. A detergent tablet according to claim 3 or 4, wherein the agent that reduces the melting point of carbamide is a salt.

6. A process for making a coated solid detergent tablet comprising the steps of

   - providing a particulate detergent composition
   - compacting said detergent composition into tablets
   - providing a coating material in its liquid phase, said coating material comprising carbamide
   - applying said coating material to at least a portion of the exterior surface of said detergent tablet
   - solidifying said coating material on said exterior surface of said tablet

7. A process according to claim 6 wherein said coating material comprises at least 50% carbamide.

8. A process according to claim 6 wherein coating material comprises carbamide and an agent that reduces the melting point of carbamide.

9. A process according to claim 8, wherein said agent that reduces the melting point of carbamide co-crystallises with said carbamide.

10. A process according to claim 8 or 9, wherein said agent that reduces the melting point of Carbamide is a salt.

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EUROPEAN SEARCH REPORT

Application Number
EP 02 01 9268

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The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	22 November 2002	Serbetsoglou, A

CATEGORY OF CITED DOCUMENTS

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EUROPEAN SEARCH REPORT

Application Number
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